SYNTHESIS AND CHARACTERIZATION OF Mo-DOPED TERNARY METAL OXIDE BASED SUPERCAPACITORS

SHORT SYNOPSIS

Submitted in partial fulfillment of the requirement of the degree of

DOCTOR OF PHILOSOPHY

by

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DECLARATION BY THE SCHOLAR

I hereby declare that the short synopsis entitled "**Synthesis and Characterization of Mo-Doped Ternary Metal Oxide based Supercapacitors**" being submitted in partial fulfillment of the requirement for the degree of Doctor of Philosophy in the Department of Science (Physics) under the School of Sciences of Manav Rachna University, Faridabad during the academic year 2024-25.

It is the work that is expected to be carried out under the supervision of **Dr. Anshuman Sahai** and co- supervision of **Dr. Deepti Maikhuri** and **Dr. Yogesh Kumar**.

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CERTIFICATE BY SUPERVISOR(S)

This is to certify that, the short synopsis entitled "**Synthesis and Characterization of Mo-Doped Ternary Metal Oxide based Supercapacitor**" is an original piece of work done by research scholar Pankaj Kumar Sharma under the supervision of Dr. Anshuman Sahai and co-supervision of Dr. Deepti Maikhuri and Dr. Yogesh Kumar for the degree of Doctor of Philosophy in the Department of Science (Physics) under the School of Sciences of Manav Rachna University, Faridabad during the academic year 2024-25.

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ABSTRACT

Electrostatic capacitors are confined to low power applications due to their low capacitance values. However, supercapacitors (SCs) can store a lot more energy than conventional capacitors and can produce more power than batteries. Researchers are now interested in SCs because of their attractive characteristics including their extended lifespan, greater value of power density, and distinctive mechanism of charge propagation, among other energy storage techniques such as batteries, fuel cells and conventional electrostatic capacitors. They are crucial in bridging the gap between traditional electrostatic capacitors and batteries. The electrochemical properties of the supercapacitor are mainly determined by the electrode material. Because of their superior electrochemical capabilities, ternary metal oxides (TMOs) have emerged as a novel class of electrode materials for SCs. Compared to binary and single-component metal oxides, TMOs have more oxidation states and perform better as supercapacitors.

Keywords: Supercapacitors, Ternary metal oxides, Clean energy.

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1.Introduction-

Energy is an essential component for the existence, survival and development of human being in society. This is the key element which fulfill the many requirement of the society [1], [2]. But the major sources of the energy are fossil fuels which are exhaustible, non-renewable and hazardous for environment also. Thus, there is a need for such energy source that can meet the escalating energy demand of the future. In last decade a lot of researches have been done on these alternative sources of energy [3]. The best alternatives to fossil fuels are certain energy storage techniques such as batteries, capacitors, fuel cells, flywheels, solar systems and SCs etc. [4]. A representation of several energy storage devices is displayed in figure-1.

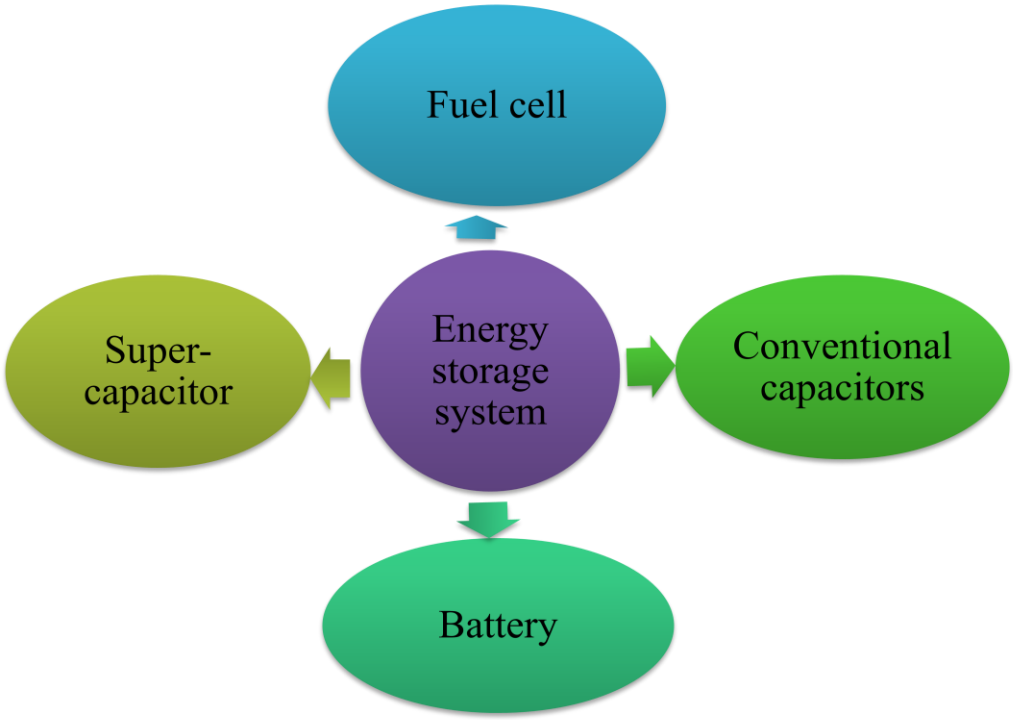

Figure-1: Representation of some energy storage devices

According to the World Energy Outlook report, currently nonrenewable sources like natural gases and coal fulfill around 68% needs, 14% of the energy needs are met by nuclear power,

15% by hydropower, and some renewable energy sources only accounts for 3% of the energy requirement as displayed below by figure-2 [5].

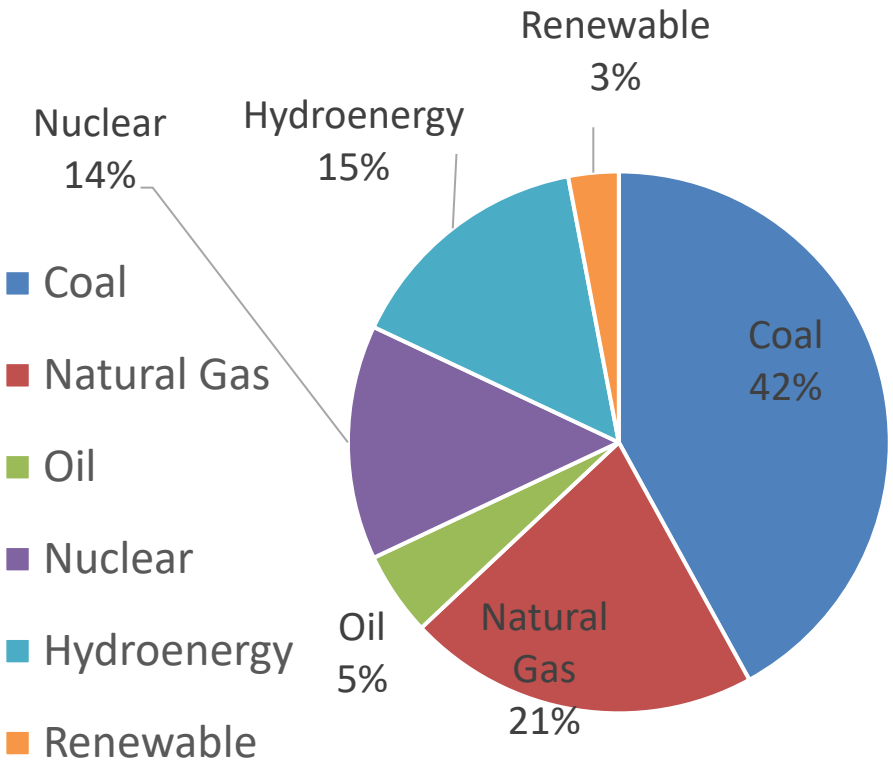

Figure-2: Distribution of energy requirement

Batteries are currently the most widely used energy storage technology in renewable energy sources, although batteries come with some disadvantages, such as a slow rate of charge and discharge, a short lifespan, large size, and hazardous for environment. Batteries are appropriate for long-term applications where a steady, gradual release of energy is required because of their greater specific energy (E_{sp})and lower specific power (slower energy discharge). Whereas, SCs come with greater specific power (faster energy discharge) but a lower energy density. They may therefore be charged and discharged considerably more quickly than batteries, but they are unable to store as much energy. Because of these characteristics, they are more suited for uses requiring rapid energy bursts and easy recharging [6]. And out of these batteries and other

sources SCs have their own significance in the energy storage [2], [7]. Power delivering capacity throughout the clock, environmental friendliness, cost effectiveness of electrode material, improved safety, little heating, device stability, lightweight design, and flexible packing of these sources makes them suitable energy storage device and future fuel [8], [9]. A SCs is consisted of two electrodes, if both the electrode materials are identical then it is known as symmetric SCs and if both the electrode materials are different then it is known as asymmetric SCs [10], [11]. Supercapacitors general schematic is displayed in figure-3.

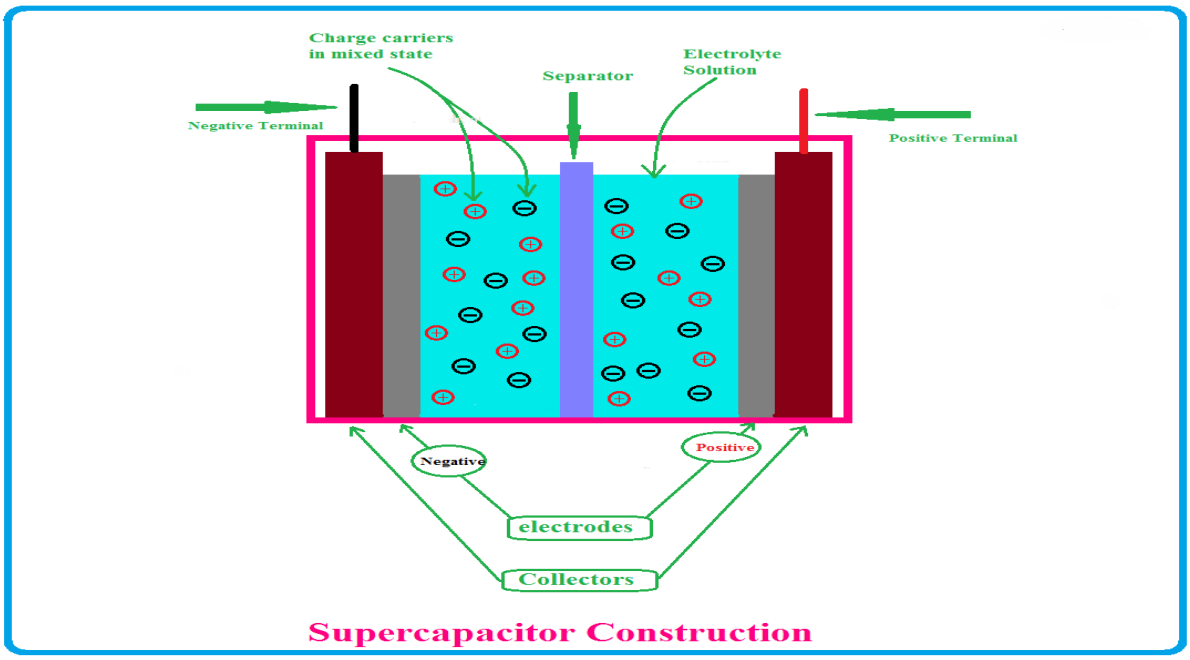

Figure-3: General schematic of Supercapacitors

The charge-discharge process at the electrode-electrolyte interface is the basis for the energy storage in SCs, which is identical to conventional capacitors [12]. The supercapacitor begins to charge when a voltage is applied across its electrodes. The oppositely charge ions begin to be attracted to both the electrodes. This means that positive charges or ions are drawn to oppositely charged terminal, similarly negative charges or ions are drawn towards oppositely charged terminal. Consequently, two different layers of oppositely charged ions are formed near both the electrodes [13]. The charging mechanism of SCs device is shown by figure-4.

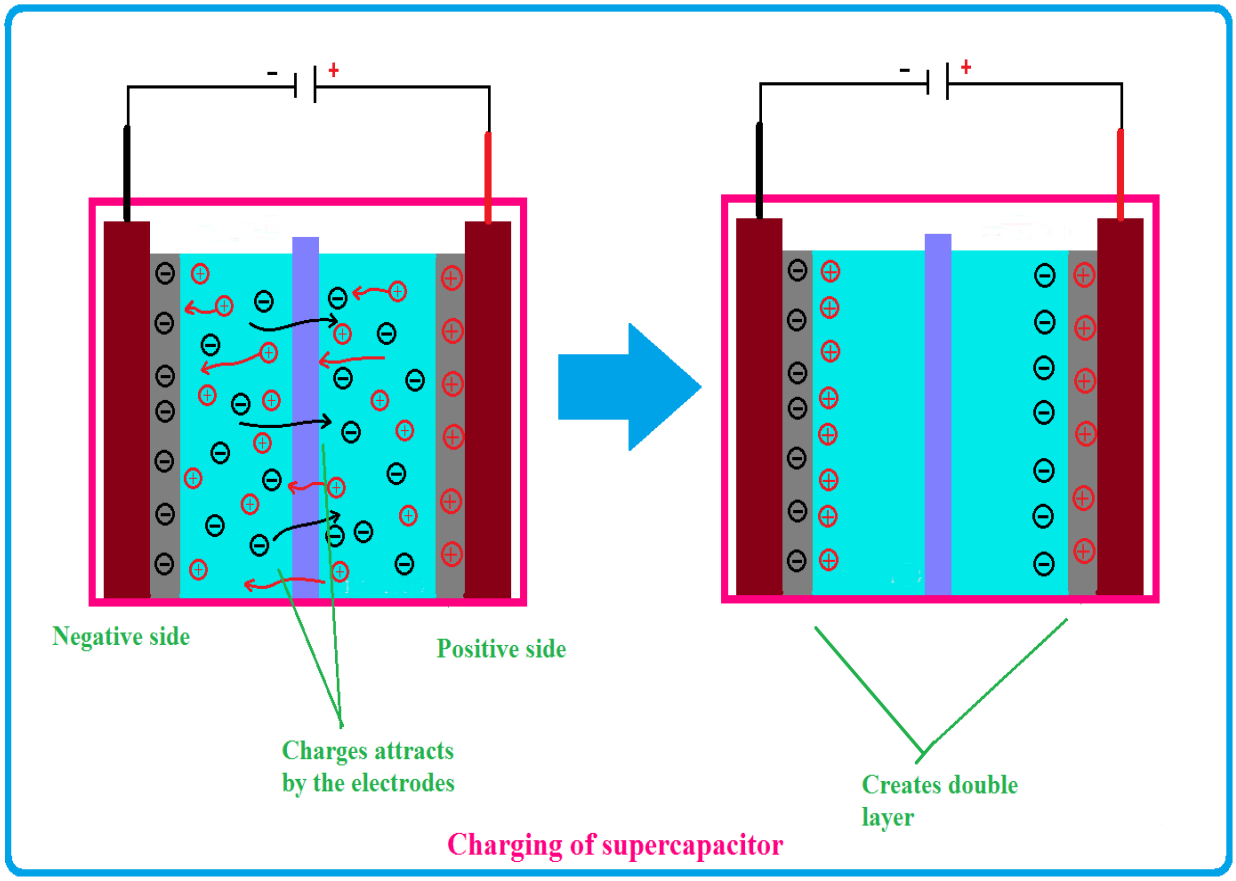

Figure-4: Charging mechanism of SCs

The ions begin dispersing into the electrolyte solutions and entering the mixed state when a load is connected across the supercapacitor. Because the electrodes are unable to pull the ions or charges for the time load is connected. The discharging mechanism of SCs device is shown by figure-5.

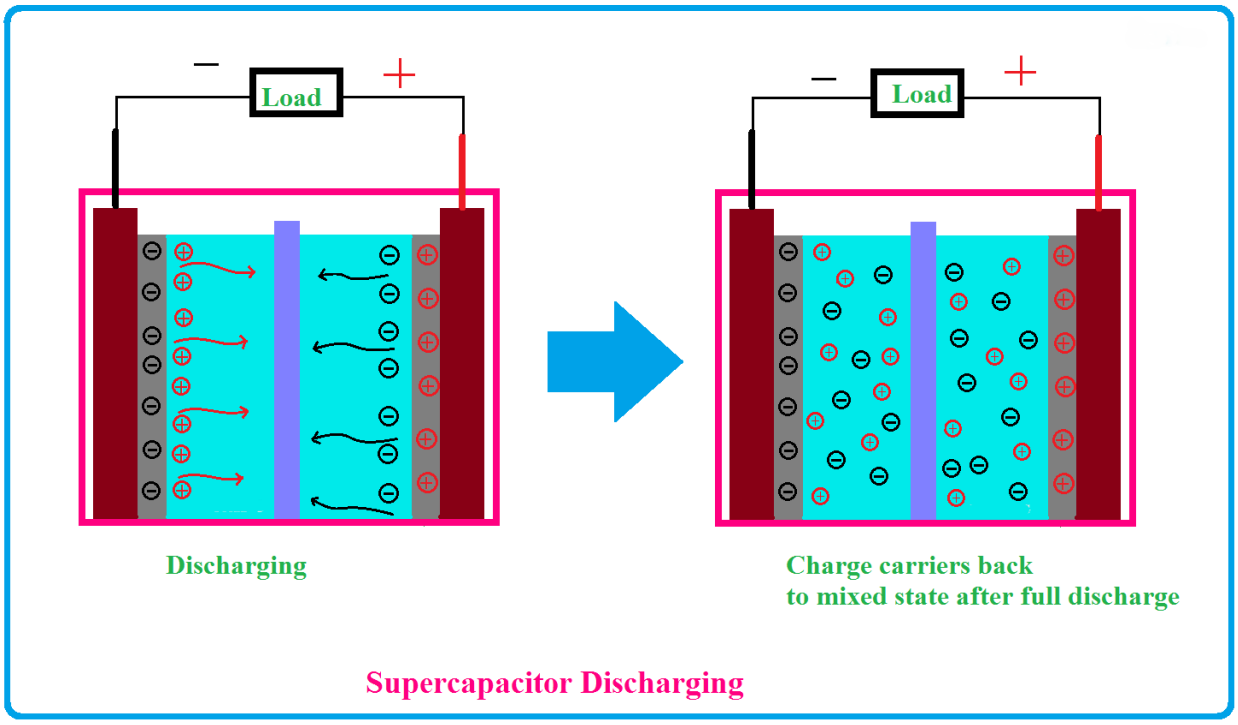

Figure-5: discharging mechanism of SCs

Considering charge storing process SCs can be classify into three different categories that includes (i) electric double layer capacitors (EDLCs) which uses non-faradic reactions in double layer electrodes to store charge electrostatically, (ii) pseudo capacitors which uses faradaic reactions to store charge electrochemically via means of the electrode and electrolyte's charge transfer and (iii) Hybrid capacitors which stores charge using both Faradaic and non-Faradaic approaches (electrostatically and electrochemically) [14], [15], [16]. Figure-6 shows the classification of SCs.

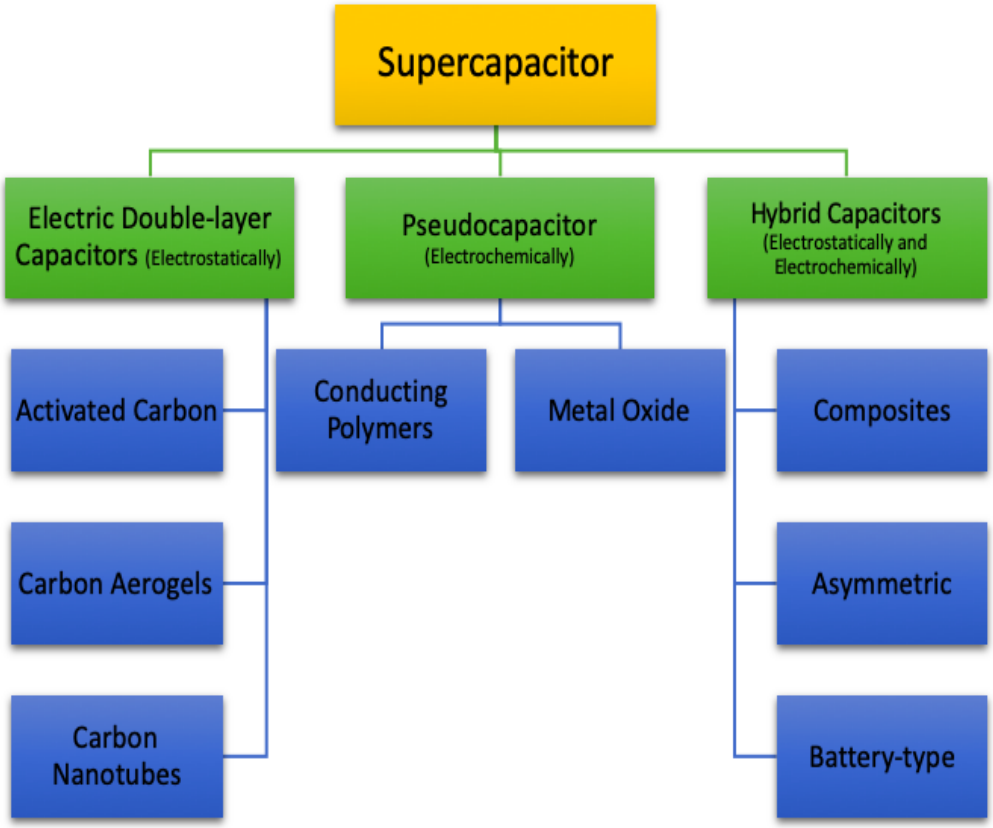

Figure-6: Classification of Supercapacitors

Recently researchers have shown their interest in ternary metal oxides (TMOs) based electrode materials for supercapacitor applications because of their advanced electrochemical properties including its long cyclic stability, quick charge-discharge rate, greater specific power, and environmental friendliness [17], [18], [19]. Due to their redox reaction process and having multiple oxidation states, metal oxides are considered to be attractive materials for electrodes in SCs. Studies show that the electrochemical performance of supercapacitors may be greatly enhanced by using electrode materials that are directly produced on conductive substrates [20], [21]. These promising characteristics of TMOs makes them suitable for energy storage devices [22]. Power density and energy density determines how well charge storing devices performs [23]. Power density, sometimes referred to as specific power (W/kg), is the ability to transmit the stored energy, while energy density E (Wh/kg) is the ability to hold the energy per unit volume

or mass [24]. Ragone plot is a practical way of evaluating functionality of any electrochemical device, which is a graph plotted between P (W/Kg) and E (Wh/Kg) [25], [23]. Figure-7 showing the graph which depict the comparison of different energy storage devices. It is evident from the graph that specific power and specific energy for SCs falls in between batteries and capacitors [24] [28].

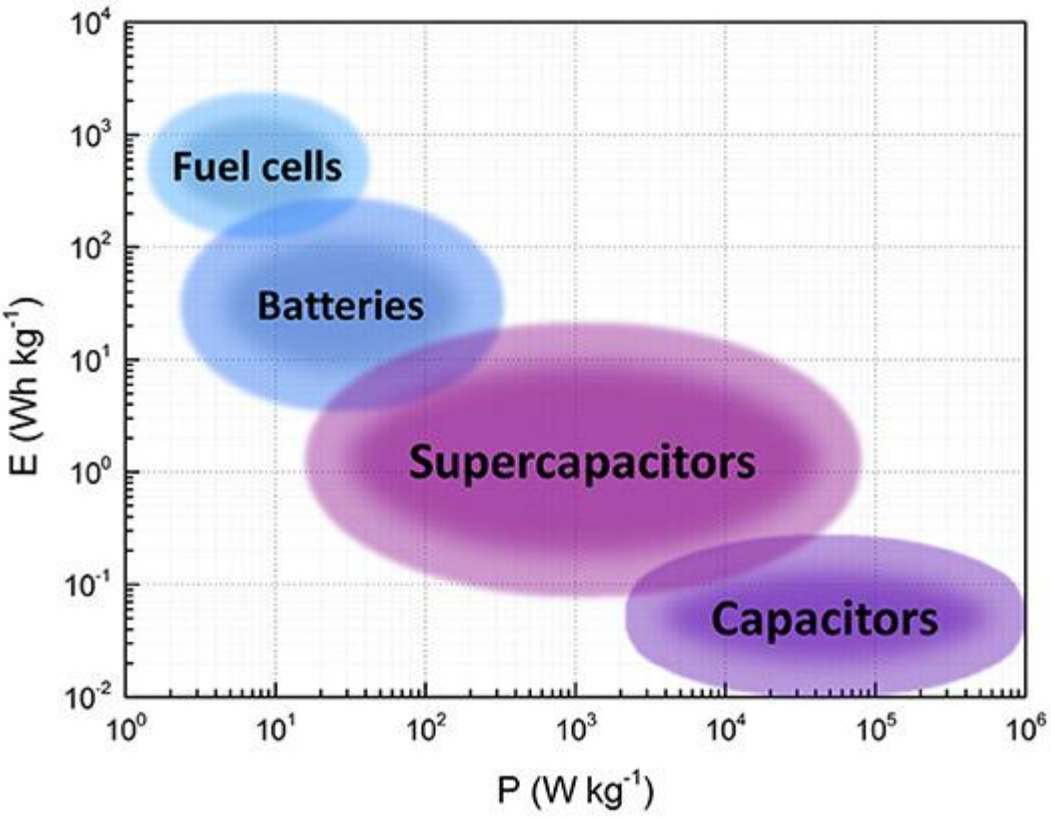

Figure-7: Comparison of some energy storing devices

2. Literature Review

2.1 History of Supercapacitors

The concept of the "Electric Double Layer" was initially proposed by Hermann Von Helmholtz, which serves as the foundation for supercapacitors in 1853[29], [30]. The first electrochemical capacitor was invented and patented in 1957 by H.I. Becker of “General Electric” under the name " "Low voltage electrolytic capacitor with porous carbon electrode" [14], [31]. Subsequently, in 1969, Tetra alkyl ammonium salt was used as an electrolyte having a high surface area carbon material electrode in the Electrochemical device, which was developed by the American Oil Company “Standard Oil of Ohio” [32], [33]. Additionally, Donald L. Boos received a patent for the gadget known as a "Electrolytic capacitor having carbon paste electrodes" in 1970 [34]. A Japanese multinational information technology company called "NEA" began marketing EDLC technology in 1978 as a supercapacitor for memory backup in computers [35]. However, it had a poor specific energy value. A novel process for storing charge was unveiled by Brain Evans Conway in 1985 [36]. This mechanism involved a redox reaction occurring in close proximity to the electrode surface [37]. These days, these gadgets are known as pseudo capacitors. RuO_2 was used in 1991 by B.E. Conway's group to create a pseudo capacitor with high specific capacitance and low series resistance [31], [38]. Since then, supercapacitor research and development have advanced quickly. To take advantage of capacitors' high power density and batteries' high energy density, the concept of storing energy through hybrid mode was developed [39]. The early supercapacitors ran at low voltages and had a low specific capacitance. For enhancing the supercapacitor's energy density, it was necessary to either create new materials or increase the electrode's surface area [40]. Many studies have been conducted with the goal of improving power and energy densities, lightening the weight of supercapacitors, and making them less harmful to the environment [16]. The development over the last 40 years is evident in the fact that modern supercapacitors may attain specific capacitance of hundreds of farads and operated at thousands of volts [41]. For their work in the field of energy storage technology, three scientists John B. Goodenough, M. Stanley Whittingham, and Akira Yoshino were awarded by the prestigious Nobel Prize of chemistry in 2019 [42], [43].

2.2 Metal oxide-based supercapacitors

- RuO_2, MnO, TiO_2, NiO, CoO, CuO, and their composite materials are potential candidate for supercapacitor [44].
- RuO_2 and MnO_2 are the prominent electrode material and have high theoretical capacitance about 1450 and 1270 F/g respectively [45].
- RuO_2 possesses high electronic conductivity of 10^4 s/cm [46].
- However, RuO_2 is extremely expensive (5000/- @1g), not earth abundant, and somewhat toxic [47].
- Tungsten, vanadium and some other metal oxides are cheap in material cost, environmentally friendly and present in large amount in the earth's crust [48], [49], [50].

Early researches in metal oxide were focused on single metal oxides based SCs. Lee and Goodenough invented first metal oxide based SCs in 1999. They utilized MnO_2 as the electrode material for this purpose and showed that these materials can be building block element for supercapacitor application[51]. Later on, synthesis was done using two different metals for this purpose. But single and binary MOs exhibit smaller specific capacitance because of less redox activities. M. Huang et al. synthesized MnO_2 based electrode material using hydrothermal method without using any surfactant. They achieved specific capacitance (Csp) 365 F/g at 0.25 A/g density of current with good retention of capacitance 90.4% after 3000 charge-discharge cycles [52]. S. Uke et al. prepared $NiCo_2O_4$ using two different metals nickel and cobalt for SCs applications using hydrothermal method with the help of surfactant. As prepared nano morphs exhibit high energy density of 21.3 Wh/kg and power density of 150 W/kg combined with an excellent specific capacitance 479 F/g [53]

In order to increase the functionality of SCs, trimetallic compound or trimetallic oxides were prepared for utilization as electrode materials and these materials showed the potential to be good electrode material having better electrochemical performances [54]. Y. Huang et al. used hydrothermal process to synthesized $Ni_xCo_yMo_zO$ (Ternary MOs) and it possess better retention of material of around 90% after 10000 charge-discharge cycles, large Mass specific Capacitance of order 867.3 F/g, higher energy density of 22.02 Wh/kg and good power density of 3.50 W/kg

[53]. Yen Sun et al. used a hydrothermal process to create ternary metal oxides based asymmetric SCs of nickel and cobalt. As prepared TMOs exhibit decent power density of 13.37 W/kg and a greater energy density of 18.92 Wh/kg. and also, better retention of material 83.2% after 10,000 cycles. These TMOs also delivers highest areal capacitance of 2.28 F/cm^2 at 5 mA/cm^2 [54].

3. Description of broad area

Based on the literature survey it is evident that primarily research have been done on single and binary metal oxides-based materials for SCs. Yet less reports are available for ternary metal oxide-based material for SCs applications. But these TMOs can play an important role in the field of energy storage [55], [56], [57]. TMOs strong redox activity, excellent theoretical capacitance, and affordable price have drawn a lot of interest of researchers [19], [60].

3.1 Applications of supercapacitors

Based on the literature review [44-50], few of the probable applications of SCs are discussed below:

- Supercapacitors are primarily used when quick charging and discharging are required.
- Digital cameras utilize supercapacitors to flash lights.
- A great tool for regenerative brakes due to fast charging and discharging.
- In defibrillators to shock people's hearts.
- Hybrid electric buses for short routes and multiple stops works with supercapacitors and batteries.
- Handle power fluctuations in grid application.
- In start-up mechanism of automobiles.
- In aerospace.
- SCs are used in various portable devices and gadgets etc.

4. Objectives of the study

The present research work proposes following objectives:

- Synthesis of Mo-doped ternary metal oxide-based nanostructures by hydrothermal reaction.
- X-ray Diffraction (XRD) will be used for structural information.
- Using Scanning Electron Microscopy (SEM), morphology will be examined.
- Fourier-transform infrared (FTIR) will be used to examine the vibrational groups.
- Fabrication of electrode for supercapacitors.
- Electrochemical characterization using Cyclic Voltammetry (CV), Galvanostatic Charge Discharge (GCD) and Electrochemical Impedance Spectroscopy (EIS).

5. Methodology to be adopted

5.1 Synthesis of Mo-doped Ternary Metal Oxide (TMOs) based electrode material for supercapacitors

The Hydrothermal method will be used to synthesize nanoparticles and then Supercapacitor will be fabricated by sandwiching Whatman filter paper between two activated electrodes. In hydrothermal method, nanomaterials are synthesized under the condition of high temperature and pressure in aqueous solvents. These nanoparticles will be highly homogeneous and of desired shape.

5.2 Characterization and Analysis

As prepared TMOs based electrode materials will be further characterized by an assortment of characterization techniques.

5.2.1 Material Characterization

- X-ray diffraction (XRD): For structural information of synthesis material.
- Scanning electron microscopy (SEM): For study about morphology of nanostructure.
- Fourier-transform infrared (FTIR): For functional group present in the synthesized material.

5.2.2 Electrochemical Characterization:

- Cyclic Voltammetry (CV): For measuring the current response that develops in the electrochemical device.
- Galvanostatic Charge Discharge (GCD): For Specific capacitance, cyclic stability, energy density and power density.

- Electrochemical Impedance Spectroscopy (EIS): For Specific capacitance (C_{sp}) and Internal resistance.

6. Expected outcomes of the research

It is expected that the synthesized TMOs based electrode material in SCs will have improved energy, power density and better cyclic stability. As prepared electrode material can be utilized in SCs to fulfill the rising energy demand throughout the clock. These electrode materials being environment friendly in nature will also help in sustainable development.

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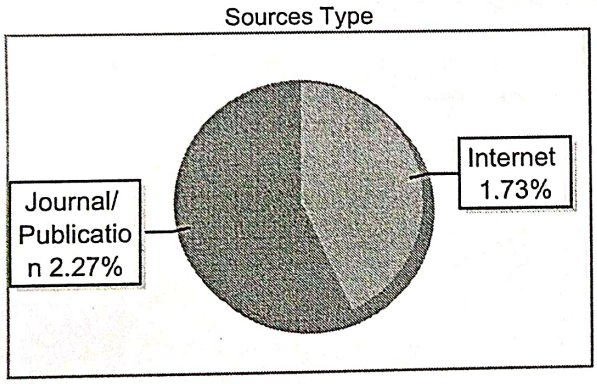

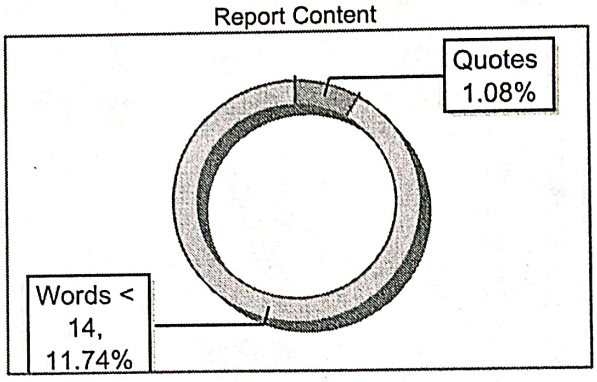

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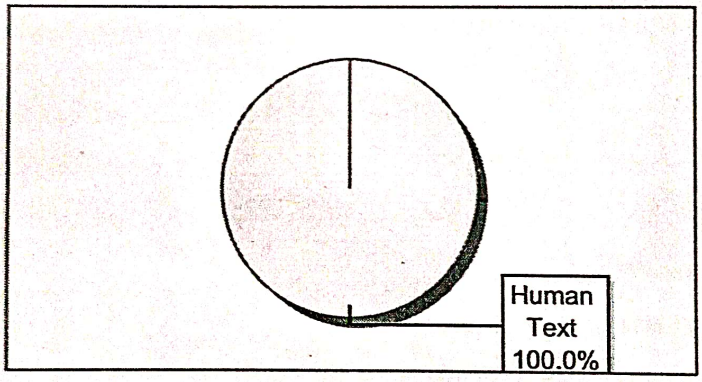

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